



Year: 2015

Effect of different surface pretreatments and adhesives on the load-bearing capacity of veneered 3-unit PEEK FDPs

Stawarczyk, Bogna ; Thrun, Hadelinde ; Eichberger, Marlis ; Roos, Malgorzata ; Edelhoff, Daniel ; Schweiger, Josef ; Schmidlin, Patrick R

Abstract: STATEMENT OF PROBLEM Polyetheretherketone (PEEK) can be used as a framework material for fixed dental prostheses (FDPs). However, information about the fracture load of veneered PEEK FDPs is still scarce. **PURPOSE** The purpose of this in vitro study was to investigate the influence of different PEEK surface pretreatments and adhesive systems on the fracture load of 2 differently veneered FDPs. **MATERIAL AND METHODS** Four hundred eighty anatomically shaped 3-unit PEEK frameworks were milled, airborne-particle abraded with 50 μ m alumina powder, and divided into 4 groups according to the following surface pretreatment (n=120): plasma treatment, etching with either sulfuric acid or piranha solution, and no further treatment. All groups were then allocated to 4 conditioning groups: visio.link, Ambarino P60, Signum PEEK Bond, or no conditioning. They were veneered with Signum Composite (n=15) or Signum Ceramis (n=15). Upon completion, the FDPs were thermally aged, and fracture loads and failure types were determined. Statistical analysis was performed with 3/2/1-way ANOVA with the post hoc Tukey HSD test ($\alpha = .05$). **RESULTS** The highest fracture loads were achieved without treatment in combination with visio.link (737 ± 138 N). The lowest values were obtained after piranha acid etching and conditioning with visio.link (277 ± 71 N); both groups were veneered with Signum Composite. The results, however, indicated no clear influence of either pretreatment or conditioning. With few exceptions, FDPs veneered with Signum Composite showed higher fracture load values compared to Signum Ceramis. After thermocycling, all FDPs showed cracks in the veneering composite resin material in the pontic region, regardless of the PEEK pretreatment or the adhesive system used. After loading, no fractures of the PEEK frameworks were evident in any FDPs, but chipping of the veneering material was observed. **CONCLUSIONS** With respect to the fracture types after thermocycling, pretreatment, conditioning, or veneering resin cement did not affect the fracture results.

DOI: <https://doi.org/10.1016/j.prosdent.2015.06.006>

Posted at the Zurich Open Repository and Archive, University of Zurich

ZORA URL: <https://doi.org/10.5167/uzh-115424>

Journal Article

Accepted Version



The following work is licensed under a Creative Commons: Attribution-NonCommercial-NoDerivatives 4.0 International (CC BY-NC-ND 4.0) License.

Originally published at:

Stawarczyk, Bogna; Thrun, Hadelinde; Eichberger, Marlis; Roos, Malgorzata; Edelhoff, Daniel; Schweiger, Josef; Schmidlin, Patrick R (2015). Effect of different surface pretreatments and adhesives on the load-bearing capacity of veneered 3-unit PEEK FDPs. *Journal of Prosthetic Dentistry*, 114(5):666-673.
DOI: <https://doi.org/10.1016/j.prosdent.2015.06.006>

Effect of different surface pretreatments and adhesives on the load-bearing capacity of veneered 3-unit PEEK FDPs

Bogna Stawarczyk, Dr biol. hum. Dipl Ing (FH) MSc CDT,^a Hadelinde Thrun, med dent,^b Marlis Eichberger, CDT,^c Malgorzata Roos, PhD,^d Daniel Edelhoff, Prof Dr med dent CDT^e, Josef Schweiger, CDT^f, and Patrick R. Schmidlin, Prof Dr med dent.^g

^aDr biol hum Dipl Ing (FH) MSc CDT, Department of Prosthodontics, Dental School, Ludwig-Maximilians University Munich, Munich, Germany.

^bmed dent, Clinic of Preventive Dentistry, Periodontology and Cariology, Center of Dental Medicine, University of Zurich, Switzerland.

^cCDT, Department of Prosthodontics, Dental School, Ludwig-Maximilians University Munich, Munich, Germany.

^dPhD, Department of Biostatistics, Epidemiology Biostatistics and Prevention Institut, University of Zurich, Zurich, Switzerland

^eProf Dr med dent CDT, Department of Prosthodontics, Dental School, Ludwig-Maximilians University Munich, Munich, Germany.

^fCDT, Department of Prosthodontics, Dental School, Ludwig-Maximilians University Munich, Munich, Germany.

^gProf Dr med dent, Department of Prosthodontics, Dental School, Ludwig-Maximilians University Munich, Munich, Germany.

Corresponding author:

Dr Bogna Stawarczyk

Ludwig-Maximilians University Munich Goethestrasse 70

80336 Munich

GERMANY

E-mail: bogna.stawarczyk@med.uni-muenchen.de

ACKNOWLEDGMENTS

The authors would like to thank nt-trading, bredent, Heraeus Kulzer, and Creamed for generously supporting this study with materials. Piezobrush Reinhausen kindly provided the plasma device for this investigation.

Effect of different surface pretreatments and adhesives on the load-bearing capacity of veneered 3-unit PEEK FDPs

ABSTRACT

Statement of problem. Polyetheretherketone (PEEK) can be used as a framework material for fixed dental prostheses (FDPs). However, information about the fracture load of veneered PEEK FDPs is still scarce.

Purpose. The purpose of this in vitro study was to investigate the influence of different PEEK surface pretreatments and adhesive systems on the fracture load of 2 differently veneered FDPs.

Material and Methods. Four-hundred-eighty anatomically shaped 3-unit PEEK frameworks were milled, airborne-particle-abraded with 50- μ m alumina powder, and divided into 4 groups according to the following surface pretreatment (n=120): Plasma treatment (A), etching with either sulfuric acid (B), or piranha solution (C), and no further treatment (D). All groups were then allocated to 4 conditioning groups: visio.link (VL), Ambarino P60 (AP), Signum PEEK Bond (SP), or no conditioning (CO). They were veneered with Signum Composite (SCO; n=15) or Signum Ceramis (SCE; n=15). Upon completion, the FDPs were thermally aged, and fracture loads and failure types were determined. Statistical analysis was performed with 3/2/1-way ANOVA with the post hoc Tukey HSD test ($\alpha=.05$).

Results. The highest fracture loads were achieved without treatment in combination with VL (737 ± 138 N). The lowest values were obtained after piranha acid etching and conditioning with VL (277 ± 71 N); both groups were veneered with SCO. The results, however, indicated no clear influence of either pretreatment or conditioning. With few exceptions, FDPs veneered with SCO showed higher fracture load values as compared with SCE. After thermocycling, all FDPs showed cracks in the veneering composite resin material in the

pontic region, regardless of the PEEK pretreatment or the adhesive system used. After loading, no fractures of the PEEK frameworks were evident in any FDPs, but chipping of the veneering material was observed.

Conclusions. With respect to the fracture types after thermocycling, pretreatment, conditioning, or veneering resin cement did not affect the fracture results.

CLINICAL IMPLICATIONS

Within the limitations of this in vitro study, the conventional veneering method with composite resin should not be used for PEEK FDPs.

INTRODUCTION

Polyetheretherketone (PEEK) is a high-temperature polymer of the family of polyaryletherketone (PAEK).^{1,2} It is a semicrystalline thermoplastic, consisting of an aromatic backbone molecular chain, interconnected by ketone and ether functional groups.³ PEEK is used in various medical applications because of its excellent chemical, mechanical, and thermal properties, expressed by its high strength combined with adequate milling and grinding properties.⁴ In dentistry, PEEK is used for interim abutments, implant-supported bars, clamp material, and dental implants.⁵ PEEK-blanks have a greyish/brown or pearl white, opaque color and are unsuitable for monolithic esthetic dental restorations, especially for the anterior region. Thus, veneering is required, but bonding to veneering composite resin materials remains a challenge because of the complex chemical structure of PEEK.

Some studies have investigated the bonding characteristics of PEEK and composite resins,^{3,4,6-14} and several studies have reported that a surface pretreatment using sulfuric acid improved the bond strength to composite resin materials.^{3,4,6,13} Some studies also assessed the bond strength of resin cements after etching with piranha solution.^{8,9,11,15} These studies,

however, reported conflicting results: While some studies observed no effect of piranha acid etching on the bond properties,^{8,9,15} one investigation reported higher bond strength, especially when applying an adhesive on airborne-particle-abraded and piranha-etched PEEK as compared with etching alone.¹¹ In addition, the influence of different adhesives on bond strengths to PEEK has also been investigated.^{7-10,12,14,15} Most studies showed that MMA-based adhesive materials were able to establish an adequate bonding to PEEK.^{7-9,12,15} One study observed the highest and most durable bond strength to PEEK after pretreatment with silica coating and conditioning with primer.¹⁶ Overall, the reported results showed comparable values with those obtained with ceramic, composite resin, or metal alloy framework materials.^{17,18}

The main problem of achieving adequate bond strengths between PEEK and composite resin still remains given the poor wetting capabilities of PEEK. To date, airborne-particle abrasion and etching still represent valuable methods of improving the wettability of PEEK.³ Plasma treatment offers another approach to raising the wettability of materials and could be an alternative approach to ensure higher bond strength to resin materials.^{19,20} Plasma is an ionized gas with essentially equal density of positive and negative charges, whereas an alternating electrical field at microwave or radio frequencies to electrodes can be applied by using the latter. A new surface layer is then built through excited molecules that will excite other species, which leads in a chemical way to an interaction with the surface.²¹

FDPs created with computer-aided design/computer-aided manufacturing (CAD/CAM) showed lower deformation and higher fracture load than pressed ones.²² A study examining nonveneered 3-unit PEEK frameworks with a rather small connector diameter of 7.4 mm² showed a deformation of the FDPs at 1200 N and fracture in the connector of the FDPs at 1385 N.³ If 400 N is considered the average maximum mastication

force in the load-bearing posterior area,²³ the laboratory results are increased by at least a factor of 2.3,²⁴ Therefore, PEEK was suggested as a material for FDPs.

The present study examined the fracture load of anatomically shaped and veneered 3-unit FDPs after different pretreatments in combination with different adhesive conditioning methods. With regard to the pretreatment regimen, no data are available regarding plasma, sulfuric acid, or piranha solution pretreatment. The null hypothesis was that the PEEK surface pretreatments, adhesive systems, and veneering materials used not influence restoration stability in terms of fracture resistance.

MATERIAL AND METHODS

A 3-unit anatomic abutment master model extending from a canine to a second premolar was made by laser sintering Co-Cr-Mo alloy (Ceramill NP L; Amann Girrbach AG). The master model was scanned to design an anatomic 3-unit FDP framework (3 Shape; Wieland+Dental). The latter displayed a 1-mm circular edge, a deep chamfer, and a concave base of the pontic. The width of the connector area was 3.2 mm and the height 4.5 mm. The thickness of the PEEK framework crown was 0.6 mm. Based on this master design, 480 standardized frameworks were milled (ZENO TEC 4030 M1; Wieland+Dental) from Dentokeep PEEK Disc (nt-trading, Lot.No: 11DK14001), airborne-particle abraded with 50- μ m alumina powder for 45 seconds at 0.2 MPa at a 45-degree angle from a distance of 10 mm (basic quattro IS, Renfert), and subsequently cleaned in an ultrasonic bath with distilled water for 5 minutes (L&R Transistor Ultrasonic T14). The study design is shown in Figure 1.

The specimens were divided into 4 pretreatment groups (n=120/group): (A) Plasma treatment using cold active inert helium gas plasma with a helium purity grade of > 99.99 (Piezobrush PZ1; Reinhausen Plasma) for 60 seconds at a pressure of 0.2 MPa from a distance of 10 mm directly before the veneering process, (B) etching with either sulfuric acid

(98%, Merck) for 60 seconds, (C) piranha solution for 30 seconds or (D) no further treatment (control group;). The piranha solution was made with sulfuric acid (98%) and hydrogen peroxide (30%) in the ratio of 3:1 immediately before the etching process (Table 1).

These pretreated groups were allocated to 4 conditioning groups (n=30/group): visio.link (bredent; Lot.No 114784), Ambarino P60 (Creamed; Lot.No 2011004057), Signum PEEK Bond (HeraeusKulzer, Bond I: Lot.No 010121, Bond II: Lot.No 010110), or no conditioning as the control group. Whereas visio.link was applied on the PEEK surface and immediately light polymerized at 220 mW/cm^2 for 90 seconds (Brelux Power Unit; bredent), Ambarino P60 was applied and left for 120 seconds. Signum PEEK Bond I was applied and vaporized for 10 seconds; thereafter, Bond II was applied and light polymerized at 225 mW/cm^2 for 90 seconds (HiLite Power; HeraeusKulzer).

A waxing was prefabricated on a PEEK framework as follows: In the middle of the pontic, a mold was formed by using a metal ball with a diameter of 6 mm placed centrally. In this way, 3 points of contact for the later placement of the FDP during the fracture load test could be created. Silicone moldings were then produced on the basis of the waxing on the metal master model, allowing for a standardized and reproducible anatomic shape performance. After the application of the veneering composite resin on the PEEK framework, the silicone molding was superimposed and excess material was removed. The metal abutment model was removed, and the application at the edges was controlled. The occlusal thickness of the veneering material was set at 1 mm.

For the veneering, the following were used: Signum Composite (HeraeusKulzer, Lot.No: 010506) or Signum Ceramis (HeraeusKulzer, Lot.No: VP 090712; n=15/subgroup). Both materials were polymerized for 90 seconds with HiLite Power (HeraeusKulzer). Afterward, the veneered FDPs were finished and polished (OPAL L; Renfert, Lot.No: 520-0001; MBH 13 200; Polirapid) by 1 blinded operator (H.T.). All FDPs were then

thermocycled (Thermocycler THE 1100; SD Mechatronik) from 5°C to 55°C with a dwell time of 20 seconds for 5000 cycles. Thereafter, FDPs were adhesively cemented on the airborne-particle abraded rigid Co-Cr-Mo alloy abutment models with Variolink II Base (Ivoclar Vivadent, Lot.No R46653) and Variolink II Catalyst (Lot.No R42290) using a standardized load of 4.9 N for 10 minutes and stored in distilled water at 37°C for 48 hours.

The FDPs were positioned and loaded (Zwick 1445; Ulm) applying a load with a ball of 6-mm diameter at the center of the pontic from the occlusal-lingual direction at a crosshead speed of 1 mm/minutes. In order to avoid force peaks, a Teflon foil with a thickness of 0.5 mm (Angst+Pfister) was placed between the pontic and the loading jig (Fig. 2). The fracture load was stopped as soon as the maximum fracture load decreased by 10% and the fracture type analyzed.

Twenty-eight specimens were prepared by cutting the PEEK blank into 10×10×3 mm pieces under water-cooling (Secotom-50; Struers). The specimens were polished (Tegamin-20; Struers) with a series of silicon carbide papers (SiC) up to P2400 under constant water-cooling for 10 seconds. Subsequently, the PEEK surfaces were airborne-particle abraded and pretreated (plasma, sulfuric acid, piranha acid, and untreated) as described above (n=6/group). Surface roughness was profilometrically examined 3× vertically/3× horizontally with a measuring track of exactly 6 mm and 0.25 mm distance between each track (M400 V3.11-11 – SD26 V1.02-15; Mahr) (n=6/group). For the assessment of surface modification after pretreatment, the specimens (n=1/group) were assessed with scanning electron microscopy (SEM) (Carl Zeiss Supra 55 VP Gemini; Carl Zeiss). The specimens (exception plasma treated) were ultrasonically cleaned for a further 5 minutes and stored in a desiccator for 7 days (Mettler U30 type with Roth Silica Gel Orange; Carl Roth) at a constant temperature of 24°C. Subsequently, the specimens were gold-palladium sputtered, and the surface was evaluated with a working distance of 4 to 7 mm at 10 kV.

The normality of data distribution was tested with the Kolmogorov-Smirnov test ($\alpha=.05$) (SPSS v22; SPSS Inc). Three-way ANOVA was used to investigate the influence of different factors (PEEK pretreatment, adhesive systems, veneering composite resin). In order to explain the 3-way interaction, 2-way ANOVA for each level of PEEK pretreatment factor was computed. In addition, the 1-way ANOVA with 1 factor containing 32 levels (4 PEEK pretreatment \times 4 adhesive systems \times 2 veneering composite resin) was used. Where necessary the Tukey HSD post hoc test was used.

RESULTS

The Kolmogorov-Smirnov showed no violation of the assumption of normality (except 1 of 32 groups; $1/32 = 3.1\% < 5\%$). The 3-way interaction (PEEK treatment versus the adhesive system versus veneering composite resin) and all 2-way interactions were significant ($P=.012$, $P<.001$, respectively) (Table 2). The 2-way interaction obtained for each level of PEEK pretreatment factor was as follows: plasma $P=.006$, sulfuric acid $P=.521$, piranha acid $P<.001$, and without pretreatment $P=.019$.

Within the Signum Composite groups, an influence of PEEK treatment was observed among conditioned groups with visio.link ($P<.001$), Ambarino P60 ($P=.048$) and nonconditioned ($P<.001$) FDPs. Among these groups, FDPs etched with piranha showed lower fracture loads than plasma treated ones. The highest fracture loads were observed for the untreated groups (Table 2). In contrast, within the FDPs conditioned with Signum PEEK Bond, no impact of surface pretreatment was observed ($P=.974$).

Within FDPs veneered with Signum Ceramis, which were conditioned with visio.link, plasma treated groups showed higher fracture loads than groups without treatment, sulfuric acid, and piranha acid etched groups ($P=.028$). The remaining veneered with Signum Ceramis groups did not affect the PEEK treatment ($P=.822$).

Signum Composite: FDPs treated with plasma ($P=.175$) and sulfuric acid ($P=.123$) had no effect on the adhesive system used. Within the piranha-etched groups, conditioning with visio.link resulted in lower fracture loads than for untreated groups ($P<.001$). Within the nonpretreated groups ($P<.001$), conditioning with Signum PEEK Bond showed the lowest fracture load values, followed by Ambarino P60. The group conditioned with visio.link presented the highest fracture load.

Signum Ceramis: Within plasma treated groups, FDPs conditioned with visio.link showed higher fracture loads as compared with the Signum PEEK Bond and no conditioned group ($P=.011$). FDPs etched with sulfuric acid ($P=.123$) and piranha solution ($P=.766$) and nonpretreated specimens ($P=.233$) had no effect on the adhesive system used.

Within the plasma pretreated groups, the main reason for interaction ($P=.006$) between the adhesive systems and veneering composite resin was an increase in the fracture load from Signum Composite to Signum Ceramis in contrast with all other tested adhesive systems where a decrease was observed. Within the sulfuric acid etched groups, no interaction ($P=.521$) was found. Signum Ceramis showed lower fracture load values than Signum Composite ($P<.001$).

Within FDPs etched with piranha acid, the main reason for interaction was a different behavior for nonconditioned PEEK surfaces where a strong decrease in fracture load between Signum Composite and Signum Ceramis was observed. In contrast, within remaining adhesive systems, no differences were found ($P>.071$). Within nonpretreated and conditioned using visio.link, Ambarino P60, or nonconditioned FDPs, showed Signum Composite higher fracture load than Signum Ceramis ($P<.001$). Among Signum PEEK Bond no impact of veneering material was found ($P=.999$).

After thermocycling, FDPs showed cracks in the veneering composite resin in the pontic region (Fig. 3A), regardless of the PEEK pretreatment or the adhesive system used.

The tested FDPs showed no fractures of the PEEK frameworks after the loading test; however, chipping of the veneering composite resin was seen (Fig. 3B).

The airborne-particle-abraded surfaces and PEEK without additional pretreatment showed the highest surface roughness values ($P<.001$). The surface roughness of the remaining pretreatment groups were in the same statistical range of values (Fig. 4). The SEM images confirm this statement (Fig. 5).

DISCUSSION

In general, all tested pretreatment methods and/or additional application of adhesives on PEEK surfaces affected the fracture load results. However, no clear pattern could be identified because of significant interactions. Therefore, the tested null hypothesis that treatment application with/without adhesives do not affect the fracture load of veneered 3-unit PEEK FDPs was rejected. Another study tested the bond strength between plasma treated PEEK surfaces and composite resins and also reported no effect on plasma treatment.⁷ However, the authors observed an improvement on bond strength after application of MMA-based adhesive materials (Signum PEEK Bond and visio.link). Studies showed that treatment using silica coating and using multifunctional acrylates containing primer resulted in durable bonding to PEEK surfaces.^{12,16} In this study, no further pretreatment after airborne-particle abrasion and adhesive treatments using visio.link in combination with Signum Composite showed the highest fracture load results. In contrast, groups etched with piranha acid and the application of visio.link led to lower fracture load values as compared with other groups. In general, PEEK has a very inert surface with a low absorption of water and is highly resistant to organic and inorganic chemicals. One study investigated the work of adhesion between different pretreated PEEK and composite resins.²⁴ The authors reported that adhesion values ranged between 95 and 108 mN/m, depending on the combination tested. However, it was

difficult to interpret which chemical bindings, on a molecular level, have been achieved between the composite resin cements and the activated PEEK surface.

For the specific use of low-density cold active inert gas plasma on small areas as in dentistry, a plasma pen was designed in the size and form of a dental handpiece.¹⁹ In general, low-pressure gas plasma shows 4 effects on surface chemistry: surface cleaning, microetching, surface activation, and ablation.²¹ One study has examined the impact of plasma with cold active inert argon gas for 20 seconds (0.2 MPa) at a distance of 10 mm on adhesion between PMMA and composite resin cements.¹⁹ Another study examined the influence of plasma pretreatment between PEEK surface and self-adhesive resin cements with the same parameters and reported no effect.⁷ In the latter study, different plasma treatment parameters were tested, such as different plasma treatment times (5 to 120 seconds), pressures (0.05 to 0.6 MPa), and different gas (argon/helium); however, no impact on the fracture load results was observed.

This investigation used 2 different veneering composite resins based on the same matrix; however, Signum Ceramis (86 % w/w) was higher filled as compared with Signum Composite (74 % w/w). In general, FDPs veneered with Signum Composite tend to result in higher fracture loads than those veneered with Signum Ceramis. A possible explanation for this is the lower viscosity of Signum Composite as compared with Signum Ceramis; this characteristic may have resulted in a better penetration of the veneering materials into the micropores created after pretreatment.

A previous study examined the fracture load of 3-unit PEEK frameworks without veneering and showed a mean fracture load of 1383 N.³ Another study tested the effect of the fabrication method of monolithic PEEK FDPs and observed fracture loads ranging between 1738 and 2354 N.²² In both studies, no aging was performed. In the present study, groups without pretreatment and adhesive application with visio.link combined with Signum

Composite showed decreased fracture loads of 737 N and even lower results of 277 N were observed after etching with piranha acid in combination with visio.link and Signum Composite. In relation to physiological mastication forces of 400 N, however, these results must be considered as being in the absolutely minimal range.²³ PEEK FDPs without veneering may exhibit an increased fracture load as compared with veneered FDPs. But since the latter remain the clinical reality and benchmark because of esthetics as previously mentioned, veneered FDPs should be assessed, especially as they are in complete contrast with standard tests with simplified geometric specimens. Using this approach, however, the fracture load represents the internal tensile stresses within the FDPs after veneering and thermal stress, as well as the bond and flexural strength of the framework together with the veneering material, which results in lower fracture load as shown by our results. Thus, development and research projects to optimize veneering methods for PEEK remain necessary.

For comparability reasons, all specimens were artificially aged with thermocycling. Volumetric changes, mechanical stress, and cracks on the bonding area, especially at the edges of the veneering can occur after thermal loading, leading to decreased bond strength values. In laboratory studies, long-term storage and thermocycling are often used to test the bonding durability.¹² All FDPs showed cracks in the veneering composite resin during the thermocycling, regardless of the pretreatment, adhesives, or veneering composite resin used. The cracks may be caused by thermal stress, due to the different elastic moduli and thermal expansion coefficients of PEEK and the veneering material.

In this study, the connector area was set at 14.5 mm², while the veneered connector area was set at 24.3 mm². As no fractures of the PEEK frameworks were observed, higher fracture loads were obtained by reducing the diameter of the connector area of the framework, leading to a thicker shift and therefore a higher fracture load of the veneering

material. Fracture load depends upon the thickness of the veneering should be examined in further studies as one investigation showed that the fracture load of ceramic FDPs depended on the connector dimensions; that is, the fracture load increased with a higher connector diameter. However, in that study, no veneering was performed.²⁷ The latter studies also used alloy abutments for the fracture load test.^{3,26} The fracture loads of FDPs was also shown to depend on the rigidity of the mounting, and the materials and elastic modulus of the abutments therefore influenced the fracture loads; that is, the higher the elastic modulus of the abutment model, the higher the fracture load. The fracture loads of FDPs decreased on rigidly mounted abutments as compared with those non-rigidly mounted.²⁷ Non-rigidly mounted abutments with an elastic modulus resembling natural teeth behave similarly to those in the clinical situation.^{28,29} Therefore, the fracture loads on dentin abutments would result in lower values, which is critical, given the already lower results of veneered FDPs as compared with anatomic PEEK restorations. A major difference of this particular study from the clinical setting is that FDPs were bonded to alloy abutments. The increased thermal conductivity of the metal will alter the thermocycling so it no longer mimics the clinical situation. Therefore, aging of the FDPs on such alloy abutments (metal retains heat or cold) would falsify the results. Therefore, we decided to age the FDPs before the cementation process to stress the material thermally. This is a limitation of our study.

CONCLUSIONS

Within the limitations of this laboratory study, the following main conclusions can be drawn:

- 1) PEEK treatment and conditioning did not increase the fracture load results.
- 2) Fracture load resistance of PEEK as a framework seems sufficient for clinical application.
- 3) The cracks during thermocycling suggest that the veneering method with conventional veneering composite resin is not appropriate for PEEK frameworks.

4) Further laboratory investigations and clinical studies and an optimization of the veneering process are necessary.

REFERENCES

1. Kurtz SM, Devine JN. PEEK biomaterials in trauma, orthopedic, and spinal implants. *Biomaterials* 2007;28:4845-69.
2. Toth JM, Wang M, Estes BT, Scifert JL, Seim HB, 3rd, Turner AS. Polyetheretherketone as a biomaterial for spinal applications. *Biomaterials* 2006;27:324-34.
3. Stawarczyk B, Beuer F, Wimmer T, Jahn D, Sener B, Roos M, et al. Polyetheretherketone- a suitable material for fixed dental prostheses? *J Biomed Mater Res B Appl Biomater* 2013;101:1209-16.
4. Schmidlin PR, Stawarczyk B, Wieland M, Attin T, Hammerle CH, Fischer J. Effect of different surface pre-treatments and luting materials on shear bond strength to PEEK. *Dent Mater* 2010;26:553-9.
5. Schwitalla A, Muller WD. PEEK dental implants: a review of the literature. *J Oral Implantol* 2013;39:743-9.
6. Sproesser O, Schmidlin PR, Uhrenbacher J, Roos M, Gernet W, Stawarczyk B. Effect of sulfuric acid etching of polyetheretherketone on the shear bond strength to resin cements. *J Adhes Dent* 2014;16:465-72.
7. Stawarczyk B, Bahr N, Beuer F, Wimmer T, Eichberger M, Gernet W et al. Influence of plasma pretreatment on shear bond strength of self-adhesive resin cements to polyetheretherketone. *Clin Oral Investig* 2014;18:163-70.
8. Stawarczyk B, Jordan P, Schmidlin PR, Roos M, Eichberger M, Gernet W et al. PEEK surface treatment effects on tensile bond strength to veneering resins. *J Prosthet Dent* 2014;112:1278-88.
9. Uhrenbacher J, Schmidlin PR, Keul C, Eichberger M, Roos M, Gernet W et al. The effect of surface modification on the retention strength of polyetheretherketone crowns adhesively bonded to dentin abutments. *J Prosthet Dent* 2014;112:1489-97

10. Stawarczyk B, Keul C, Beuer F, Roos M, Schmidlin PR. Tensile bond strength of veneering resins to PEEK: impact of different adhesives. *Dent Mater J* 2013;32:441-8.
11. Hallmann L, Mehl A, Senero N, Hämmerle CHF. The improvement of adhesive properties of PEEK through pre-treatments. *Appl Surface Sci* 2012;258:7213-8.
12. Kern M, Lehmann F. Influence of surface conditioning on bonding to polyetheretherketon (PEEK). *Dent Mater* 2012;28:1280-3.
13. Sproesser O, Schmidlin PR, Uhrenbacher J, Eichberger M, Roos M, Stawarczyk B. Work of adhesion between resin composite cement and PEEK as a function of etching duration with sulfuric acid and its correlation with bond strength values. *Inter J Adhesion Adhesives* 2014;54:184-90.
14. Rosentritt M, Preis V, Behr M, Senero N, Kolbeck C. Shear bond strength between veneering composite and PEEK after different surface modifications. *Clin Oral Investig* 2015;19:739-44.
15. Keul C, Liebermann A, Schmidlin PR, Roos M, Sener B, Stawarczyk B. Influence of PEEK surface modification on surface properties and bond strength to veneering resin composites. *J Adhes Dent* 2014;16:383-92.
16. Fuhrmann G, Steiner M, Freitag-Wolf S, Kern M. Resin bonding to three types of polyaryletherketones (PAEKs)-durability and influence of surface conditioning. *Dent Mater* 2014;30:357-63.
17. Sabatini C, Patel M, D'Silva E. In vitro shear bond strength of three self-adhesive resin cements and a resin-modified glass ionomer cement to various prosthodontic substrates. *Oper Dent* 2013;38:186-96.
18. Bahr N, Keul C, Edelhoff D, Eichberger M, Roos M, Gernet W et al. Effect of different adhesives combined with two resin composite cements on shear bond strength to polymeric CAD/CAM materials. *Dent Mater J* 2013;32:492-501.

19. Liebermann A, Keul C, Bahr N, Edelhoff D, Eichberger M, Roos M et al. Impact of plasma treatment of PMMA-based CAD/CAM blanks on surface properties as well as on adhesion to self-adhesive resin composite cements. *Dent Mater* 2013;29:935-44.
20. Ourahmoune R, Salvia M, Mathia TG, Mesrati N. Surface morphology and wettability of sandblasted PEEK and its composites. *Scanning* 2014;36:64-75.
21. Liston E. Plasma treatment for improved bonding: A review. *J Adhesion* 1989;30:199-218.
22. Stawarczyk B, Eichberger M, Uhrenbacher J, Wimmer T, Edelhoff D, Schmidlin PR. Three-unit reinforced polyetheretherketone composite FDPs: Influence of fabrication method on load-bearing capacity and failure types. *Dent Mater J* 2015;34:7-12.
23. Helkimi E, Carlson G, Helkimo M. Bite force and state dentition. *Acta Odontol Scand* 1976;35:297-303.
24. Sproesser O, Schmidlin PR, Uhrenbacher J, Eichberger M, Roos M, Stawarczyk B. Work of adhesion between resin composite cements and PEEK as a function of etching duration with sulfuric acid and its correlation with bond strength values. *Int J Adhesion Adhesives* 2014;54:184-90.
25. Ambre MJ, Aschan F, Vult von Steyern P. Fracture strength of yttria-stabilized zirconium-dioxide (Y-TZP) fixed dental prostheses (FDPs) with different abutment core thicknesses and connector dimensions. *J Prosthodont* 2013;22:377-82.
26. Stawarczyk B, Ender A, Trottmann A, Ozcan M, Fischer J, Hammerle CH. Load-bearing capacity of CAD/CAM milled polymeric three-unit fixed dental prostheses: Effect of aging regimens. *Clin Oral Investig* 2012;16:1669-77.
27. Scherrer SS, de Rijk WG. The fracture resistance of all-ceramic crowns on supporting structures with different elastic moduli. *Int J Prosthodont* 1993;6:462-7.

28. Goncu Basaran E, Ayna E, Vallittu PK, Lassila LV. Load-bearing capacity of handmade and computer-aided design--computer-aided manufacturing-fabricated three-unit fixed dental prostheses of particulate filler composite. *Acta Odontol Scand* 2011;69:144-50.
29. Keulemans F, Lassila LV, Garoushi S, Vallittu PK, Kleverlaan CJ, Feilzer AJ. The influence of framework design on the load-bearing capacity of laboratory-made inlay-retained fibre-reinforced composite fixed dental prostheses. *J Biomech* 2009;42:844-9.

TABLES

Table 1. Summary of tested materials, manufacturers, and compositions.

Material	Product Name	Manufacturer	Composition
PEEK	Dentokeep PEEK	nt-trading	Polyetheretherketone, 20 wt% titanium oxide
Pre-treatment	Plasma	Piezobrush	Helium gas plasma
	Piezobrush		
	Sulfuric acid	Merck	98% sulfuric acid
	Hydrogen peroxide for mixture of piranha solution	Apotheke Innenstadt, Ludwig-Maximilians University, Munich, Germany	30% hydrogen peroxide, medically pure, stabilized
Adhesive system	visio.link	breident	Methylmethacrylate, pentaerythritol triacrylate, photo initiators
	Ambarino P60	Creamed	Dimethacrylate based on phosphonic and phosphoric acid esters
	Signum PEEK Bond	Heraeus Kulzer	Bond I: bifunctional molecules based on phosphoric acid esters and thiol compounds Bond II: methylmethacrylate, polymethylmethacrylate, photo initiators
Veneerin	Signum	Heraeus Kulzer	Multifunctional methacrylic acid esters, photo

g composit e resin	Composite Dentine A3		<p>initiators, stabilizing agents, inorganic pigments</p> <p>Fillers: 74 wt%: silicon dioxide, rheologically active type of silicon dioxide, splitted pre-polymer</p>
	Signum Ceramis Dentine A3		<p>Multifunctional methacrylic acid esters, photo</p> <p>initiators, stabilizing agents, inorganic pigments</p> <p>Fillers: 86 wt%: silicon dioxide, inorganic fillers</p>

Table 2. Three-way ANOVA results for comparison of fracture load with different pretreatment methods, use of different adhesive systems, and veneering with different composites resin.

	sum of squares	Df	Mean squares	F	P
constant parameters	97028428	1	97028428	5885	<.001
PEEK treatment	1501985	3	500662	30	<.001
adhesive system	316032	3	105344	6	<.001
veneering composite resin	858863	1	858863	52	<.001
PEEK pretreatment × adhesive system	870613	9	96735	6	<.001
PEEK pretreatment × veneering composite resin	404981	3	134994	8	<.001
adhesive system × veneering composite resin	360419	3	120140	7	<.001
PEEK pretreatment × adhesive system × veneering composite resin	353672	9	39297	2	.012
error	7386414	448	16488		
total	109081406	480			

Table 3. Descriptive statistics of fracture load [N] for each tested group.

	Adhesive system	Signum Composite		Signum Ceramis	
		mean (SD)	95% CI	mean (SD)	95% CI
with plasma	visio.link	487 (141) ^a	(407;565)	558 (132) ^b	(483;631)
	Ambarino P60	552 (133) ^a	(477;626)	406 (93) ^{ab}	(353;458)
	Signum PEEK Bond	489 (146) ^a	(407;571)	427 (122) ^a	(357;495)
	without	515 (105) ^a	(456;574)	406 (120) ^a	(338;473)
with sulfuric acid	visio.link	425 (61) ^a	(389;459)	371 (175) ^a	(273;469)
	Ambarino P60	469 (121) ^a	(401;537)	323 (115) ^a	(257;387)
	Signum PEEK Bond	464 (99) ^a	(408;519)	393 (115) ^a	(328;457)
	without	511 (150) ^{a*}	(426;594)	420 (150) ^a	(335;504)
with piranha solution	visio.link	277 (71) ^a	(237;317)	398 (133) ^a	(323;473)
	Ambarino P60	383 (125) ^{ab}	(313;453)	335 (112) ^a	(271;397)
	Signum PEEK Bond	393 (77) ^{ab}	(348;436)	399 (157) ^a	(310;487)
	without	472 (79) ^b	(427;517)	341 (112) ^a	(277;403)
without pre-treatment	visio.link	737 (138) ^c	(659;814)	513 (152) ^a	(428;597)
	Ambarino P60	562 (205) ^{ab}	(447;676)	382 (128) ^a	(310;454)
	Signum PEEK Bond	421 (168) ^a	(327;515)	389 (124) ^a	(319;459)
	without	713 (127) ^{bc}	(642;784)	457 (108) ^a	(395;517)

^{a,b} shows significant differences according to 1-way ANOVA, followed by post hoc Scheffé test between conditioning groups within one veneering resin and one pre-treatment group, separately

* nonnormally distributed groups

LEGENDS

Fig. 1. Study design, division of specimens according to different pretreatments, veneering composite resin, and adhesive systems.

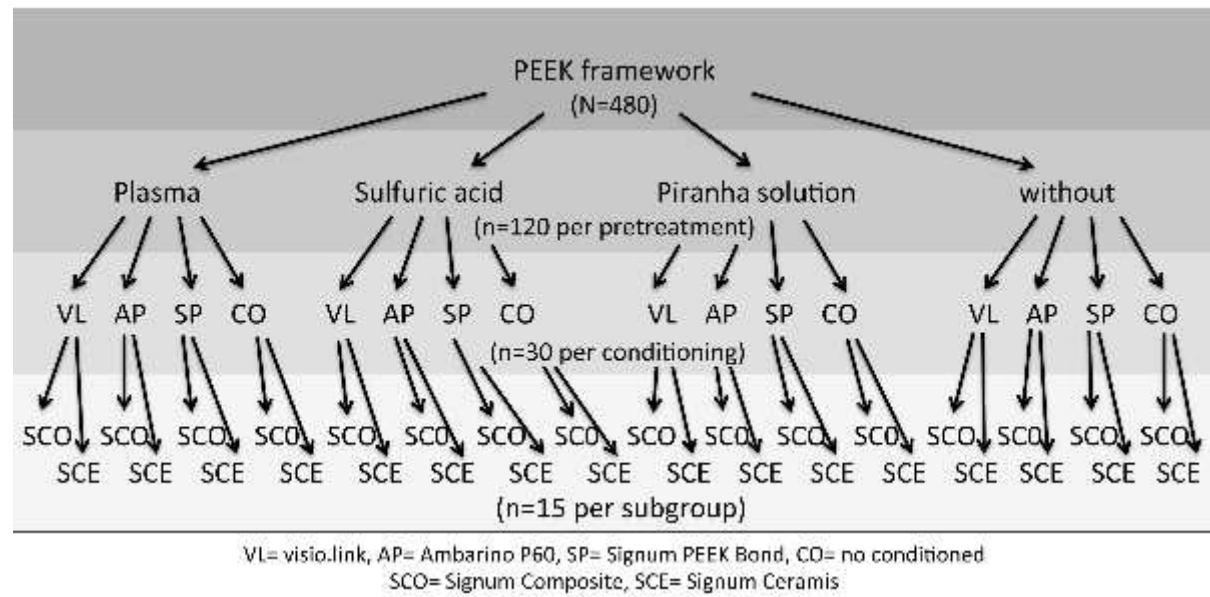


Fig. 2. Veneered PEEK FDPs positioned in universal testing machine with Teflon foil between pontic and loading jig.



Fig. 3. Failure types. A, After thermal cycling. Cracks in veneering composite resin. B, After fracture load measurements. Adhesive failures between PEEK framework and veneering composite resin.

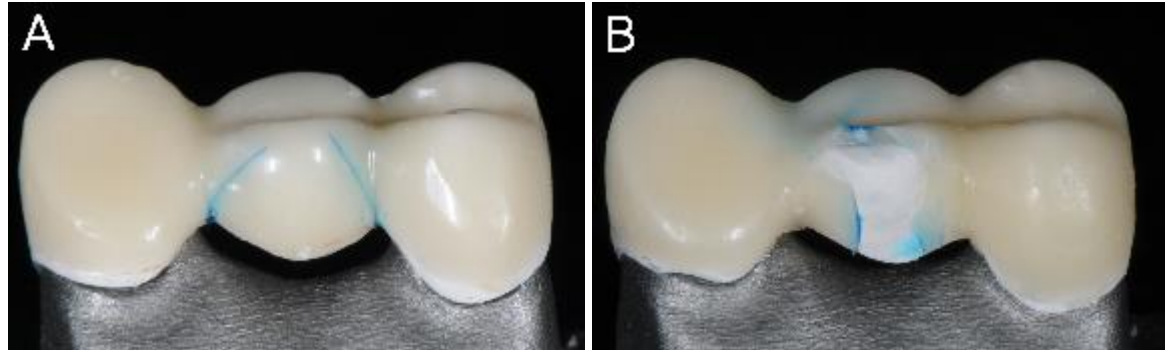


Fig. 4. Surface topography. A, After plasma treatment. B, Etched with sulfuric acid. C, Etched with piranha solution. D, Untreated PEEK surface ($\times 50\,000$ magnification).

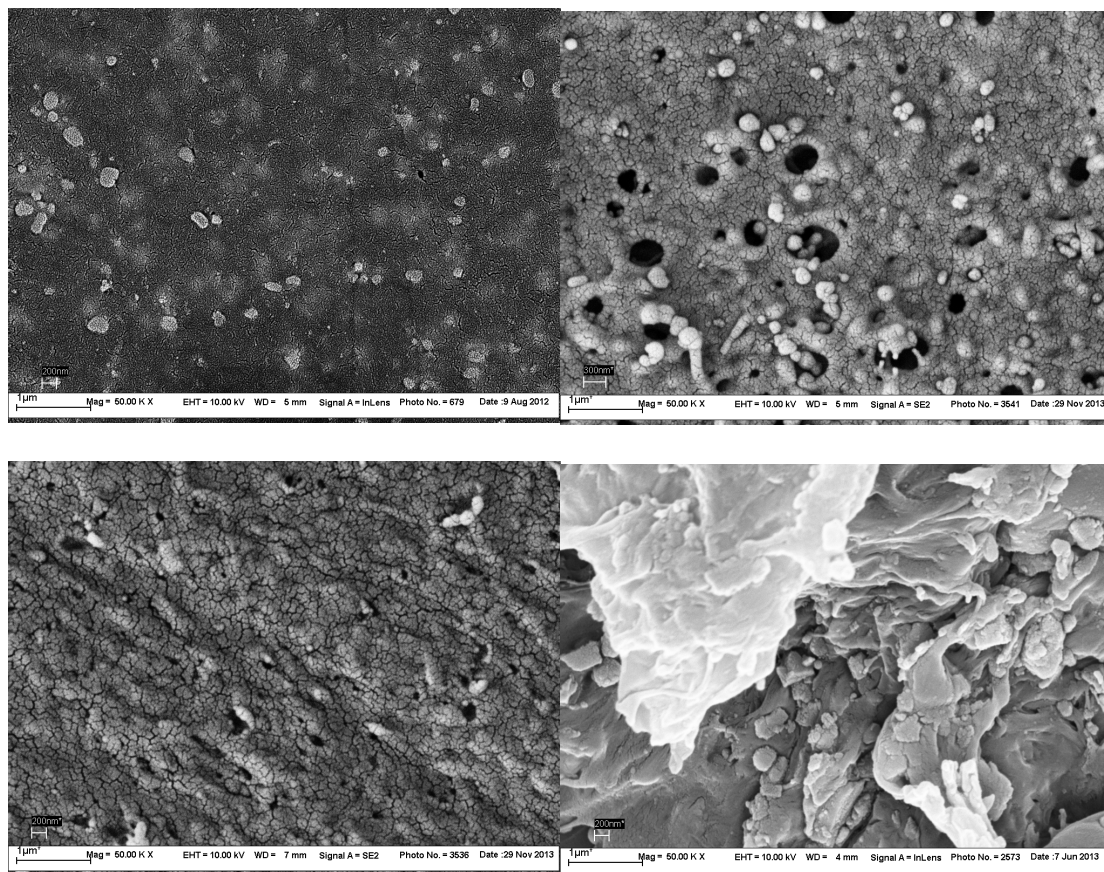


Fig. 5: Boxplot of surface roughness values after different pretreatments of airborne-particle abraded PEEK surfaces.

